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PRODUCTION OF NANO SILICA USING SOL GEL METHOD BY VARYING THE CONCENTRATION OF SURFACTANT AND TO STUDY ITS BEHAVIOUR IN BLENDED MATERIAL

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Abstract *- The spherical particles of Nano silica (NS) with controllable size have been synthesized by using tetraethoxysilane, as a starting material, with ethanol solvent and ammonia as a base catalyst using sol gel method. The method is carried out as a trial and error process by varying the concentration of surfactants viz., 2.2g, 2.4g, 2.5g, 2.6g, and 2.8g. The concentration of pH maintained as 8. The result showed that the size of NS increased the concentration surfactant also increased but the size of NS is lesser for the concentration of surfactant 2.5g comparatively other. The comparatively study of normal cement paste, silica fume varied contents (0.2%, 0.4%, 0.6%, 0.8%, 1%, 2.5%, 5%, 7.5% and 10%) and NS varied contents (0.2%, 0.4%, 0.6%, 0.8%, and 1%). The experimental results of these studies showed that the compressive strength of fresh paste was increased by increasing the content of NS up to 1% in cement paste. The setting time of cement paste was decreased by increasing the content of NS. The nature and morphology of synthesized nano silica was investigated by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and x-ray diffraction (XRD).*

Keywords: *nanoparticles, NS, NCP, SFCP, NSCP, XRD, SEM and TEM.*

1. INTRODUCTION

Concrete is the most commonly used material for construction and it consumes almost the total cement production in the world. The use of large quantities of cement produces an increasing CO_2 emission and as a consequence, increasing the greenhouse effect. The silica fines with high potential are used for replacing cement in concrete. At present, a wide range of silica products are manufactured industrially for various applications. The Worldwide demand for speciality silica's is precipitated silica, fumed silica, silica gel and silica sol etc. in this present study synthesis of nano silica using sol gel method and study its behaviour in blended material. The methods available for the preparation of Nano silica are precipitation method, olivine process, sol gel process, micro emulsion method, flame hydrolysis etc. The sol gel process is chosen because it is simple and cost effective, as it is several advantages such as synthesis may be carried out at low temperature, desired pH to yield high purity and controlled size of particles.

Bogush et al [1]. Have done extensive work on preparation of silica nano particles ranging from few hundred nanometres to several micrometres by control hydrolysis of TEOS in ethanol. Preparation of silina nano particles and its beneficial role in cementitious material is documented in research papers [2, 3] spherical silica nano particles (n-sio_2) with controllable size have been synthesized using tetra ethoxysilane as a starting material and ethanol as solvent by sol gel method. Increase in chain length of surfactant and solvent resulted in decreasing particle size of silica nanoparticles. The size silica particle was also controlling use NH_3 as base catalyst. addition of silica nanoparticles in to cement paste improved the microstructure of the paste and calcium leaching is significantly reduced as n-sio_2 reacts with calcium hydroxide and form additional calcium silicate hydrate (C-H-S) gel. The experimental result of these studies showed that compressive strength of fresh cement pastewas increased by increasing the content of NS upto 5%. In 1968 Stober and Fink developed a system of chemical reaction which controlled the growth silica particles. Htr importance and advantages of mono-dispersed nanometre-sized particles were shown not only scientifically, but also in various industrial applications [4]. S.tabatabaei et al. nano silica were synthesised by chemical methods. Spherical silica particles with a very narrow particle size distribution have been synthesised by the hydrolysis reaction of TEOS in ethanol containing water and ammonia. Different solvent have different effect on the size of the silica particles. Using methanol and ethano-glycerol, a stable sol stable sol could easily be obtained [5]. Effect of AlF_3 production waste on the properties of hardened cement paste obtained pure silica in research paper [5]. In this study Alf_3 production waste was investigated as the basic ingredient of a new pozzolanicmaterial. The goal of this study is to investigate the possibilities of using Alf_3 production waste, washed in ammonia solution, in cement stone specimens. Chemically treated silica gel additive was proved to reduce the amount of $Ca(OH)_2$ and $CaCO_3$ in hardened cement paste samples. Experimental research has revealed that the density in hydrated samples reduces from 2220 kg/m^3 to 2030 kg/m^3 with the increase of silica gel content from 0% to 35%. The compressive strength of samples containing 10% of silica gel additive increased by 8.04% compared to the samples without the additive [6]. Alirezanajigivi et al. have studied the effect of two different types of sio_2 (N and M series) with different ratios on the workability and compressive strength of developed binary blended concretes cured water and lime solution. The N and M series cwith on average size of 15 nm were used as obtained from suppliers. Fresh and hardened concretes incorporating 0.5%, 1%, 1.5% and 2% of N and 2% M series with constant water to binder ratio and aggregate content were made and tested. Fresh concrete test results showed that workability of binary

was reduced in the presence of both types of sio_2 nanoparticles. 1% of N series replacement with water curing improved compressive strength significantly. The ultimate strengths of binary blended concretes were gained at 2.0 wt. % replacement of cement by both series of after curing in lime solution [7].The purpose of present study is to investigate varying the concentration of surfactant and their effects in preparing silica nanoparticle and to study its behaviour in blended material with compared to silica fume.

2. EXPERIMENTAL WORK

TEOS, NH_4OH aqueous solution and ethanol (EtOH) were used and the water used for the sample preparation was purified both ion-xchange and distillation. The non- ionic surfactant for sorbitantmonostearate used for control the size Nano particle. The spherical particles of Nano silica (NS) with controllable size have been synthesized by using tetraethoxysilane, as a starting material, with ethanol solvent and ammonia as a base catalyst using sol gel method. The method is carried out as a trial and error process by varying the concentration of surfactants viz., 2.2g, 2.4g, 2.5g, 2.6g, and 2.8g. The concentration of pH maintained as 8. TEOS 1 ml, ethanol 10 ml, non-ionic surfactant 2.5g and distilled water 3 ml taken into reaction container. The solution mixed by magnetic stirrer with a constant temperature of 30^oC up to 30 mints. The sample continuously stirred without temperature up to 30 min. through added ammonia solution drop wise to control pH 8. Stirring was continued for further 2 h to get white turbid powder. It was dried overnight and then calcined in air for a period of 1h at 650^oC. The consistency and setting time of cement paste was fount for normal cement paste according to is 4301, by adding silica fume for replacement of cement in to 0.2%, 0.4%, 0.6%, 0.8%, 1%, 2.5%, 5%, 7.5% and 10% and also replacement of cement with sio_2 nanoparticles in to 0.2%, 0.4%, 0.6%, 0.8%, and 1%. Consistency was ascertained by putting the cement paste in a mould consisting of a steel ring (40 mm in height) on a sheet of glass and by determining the penetration depth of plunger applied to the top surface of the paste specimen. The initial and final setting time of fresh cement paste was determined by using vicat apparatus. The fresh cement paste with varying composition of NS and silica fume above same were casted with moulds (25 X 25 x25 mm) to prepare specimens for the measurement of compressive strength. After being demoulded at the age of one day, all specimens were cured with water and three cubes were tested at a given age of (1, 3, 7, & 28 days) using UTM.
The prepared NS were characterised with scanning electron microscope (SEM), powder X-diffraction (XRD) and transmission electron microscope (TEM).

3. RESULT AND DISCUSSION

The yields of silica nanosphere synthesised with TEOS at different concentration of surfactant as shown in XRD graph 1, 2, 3, 4 and 5

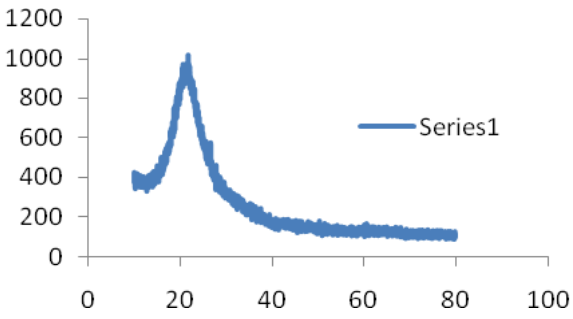

Fig.1 Concentration of surfactant

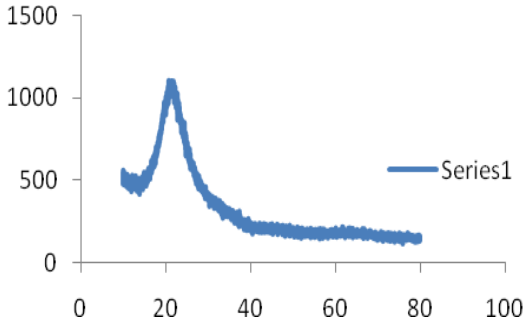

Fig.2 Concentration of surfactant

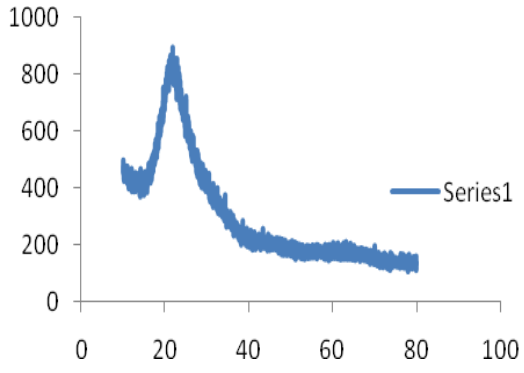

Fig.3 Concentration of surfactant

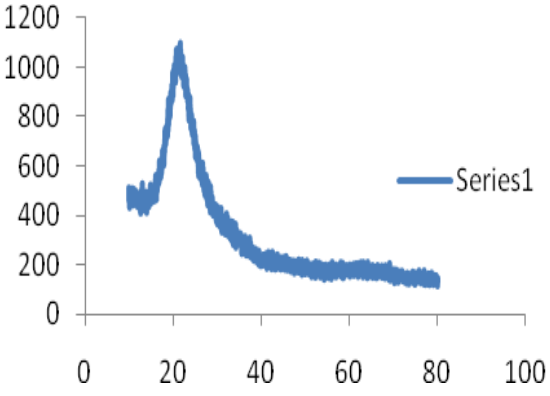

Fig.4 Concentration of surfactant

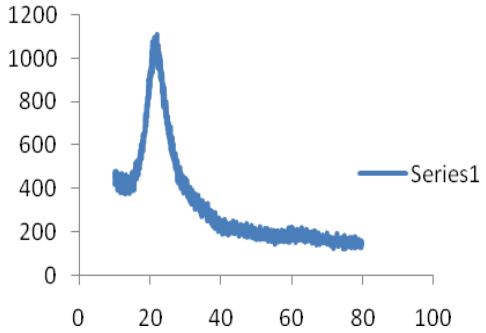

Fig.5 Concentration of surfactant

3.1 Calculation of particle size

The particle size of the silica has been determined from the X-ray diffraction data using Debye Sherrer's formula,

$D = 0.9\lambda / \beta \cos\theta$ in A^o

Where,

D -particle size

λ -wavelength of the incident X-ray beam

β- full width at half maximum of the X-ray diffraction peaks

θ -Bragg angle of X-ray diffraction peak.

The average particle size of the prepared silica is

- Surfactant 2.2g- 12.5 nm
- Surfactant 2.4g - 12.7 nm
- Surfactant 2.5g- 12.1 nm
- Surfactant 2.6g- 13.2 nm
- Surfactant 2.8g– 13.8 nm

3.2 SEM result

The SEM analysis result for the sample with the concentration of surfactant as 2.5 g is shown in fig.

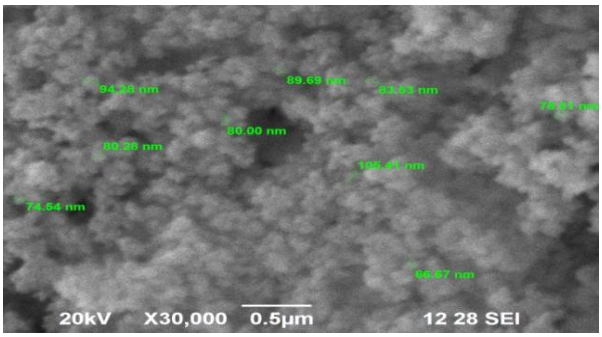

Fig.7 EDAX analysis

3.3 TEM result

Form the above results the average size of the Nano silica is 20nm-88nm. The prepared silica nanoparticles were incorporated into the cementitious system to improve mechanical properties of cement system. The silica fume was incorporated in to cementitious system to improve mechanical properties of cement paste. The Nano cement paste has been higher mechanical strength compared with normal cement and silica fume cement paste. The initial and final setting time of cement was reduced when increase the content of Nano silica. The consistency of cement paste increasing also increase the content of nanoparticles. The following tables show that consistency, initial and final setting time of NCP, SFCP, and NSCP.

Table.1

Consistency and setting times of NCP,SFCP and NSCP as shown in table

Cement paste	SF in cement (w%/w)	NS in cement (w%/w)	Consistency in %	Setting time	
				Initial in min	Final in min
NCP	-	-	28	68	218
SFCP	0.2	-	28.5	110	336
SFCP	0.4	-	27.5	98	258
SFCP	0.6	-	27	66	244
SFCP	0.8	-	27	52	218
SFCP	1	-	26.5	47	206
NSCP	-	0.2	27.5	88	308
NSCP	-	0.4	27	54	228
NSCP	-	0.6	28.5	46	207
NSCP	-	0.8	29	42	198
NSCP	-	1	29.5	38	186

Table.2

The compressive strength of NCP, SFCP and NSCP as shown in table

Cement paste	SF in cement(w%/w)	NS in cement(w%/w)	w/c	Compressive strength in N/mm^2		
				1 d	3 d	7 d
NCP	-	-	0.28	27.2	37.3	56.4
SFCP	0.2	-	0.285	30.9	49.6	46.9
SFCP	0.4	-	0.275	24.0	42.1	40.5
SFCP	0.6	-	0.27	34.1	51.2	43.2
SFCP	0.8	-	0.27	30.9	54.4	43.7
SFCP	1	-	0.265	30.4	53.8	42.6
NSCP	-	0.2	0.275	36.2	47.0	-
NSCP	-	0.4	0.27	26.4	46.4	-
NSCP	-	0.6	0.285	37.6	-	-
NSCP	-	0.8	0.29	38.4	-	-
NSCP	-	1	0.295	44	-	-

3.3 Comparison results of compressive strength

The consistency value increasing by increase the content of nanoparticle and silica fume up to 1% in cement. Initial and final setting time value decreasing by increase the content of nanoparticle up to 1% in cement was comparative normal and silica fume cement paste. Compressive strength of nanoparticle comparative was higher than the normal cement paste and silica fume cement paste. When the nanoparticles added in cementitious material was increase mechanical property 61.7% at one day, 50% at 3 day and 20.7% at7day.

4. CONCLUSIONS

Dispersed, amorphous, spherical silica Nano particles (20 nm-88 nm) can be prepared by the hydrolysis reaction of TEOS in ethanol using surfactant and ammonia as base catalyst by sol-gel method. The average particle size 20 nm- 88 nm observed from SEM and TEM analysis.

The particle size of NS can be controlled by using varying the concentration of surfactant and maintain the pH 8. The addition of nanoparticles significantly improves the mechanical properties of the cementitious material. But also reduce the setting time cement by increasing content of NS up to 1%.

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